# NATIONAL BUREAU OF STANDARDS REPORT

9460

QUARTERLY STATUS REPORT FOR THE
QUARTER ENDING OCTOBER 31, 1966 ON

NBS PROJECT 3120445

INVESTIGATION OF THE DIRECTIONAL EFFECTS
IN THE STRESS CORROSION OF ALUMINUM ALLOYS

By

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and
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for

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama

Contract H-2151A
Control 1-6-54-01046-01 (1F)

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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# NATIONAL BUREAU OF STANDARDS REPORT

**NBS PROJECT** 3120445

December 13, 1966

**NBS REPORT** 9460

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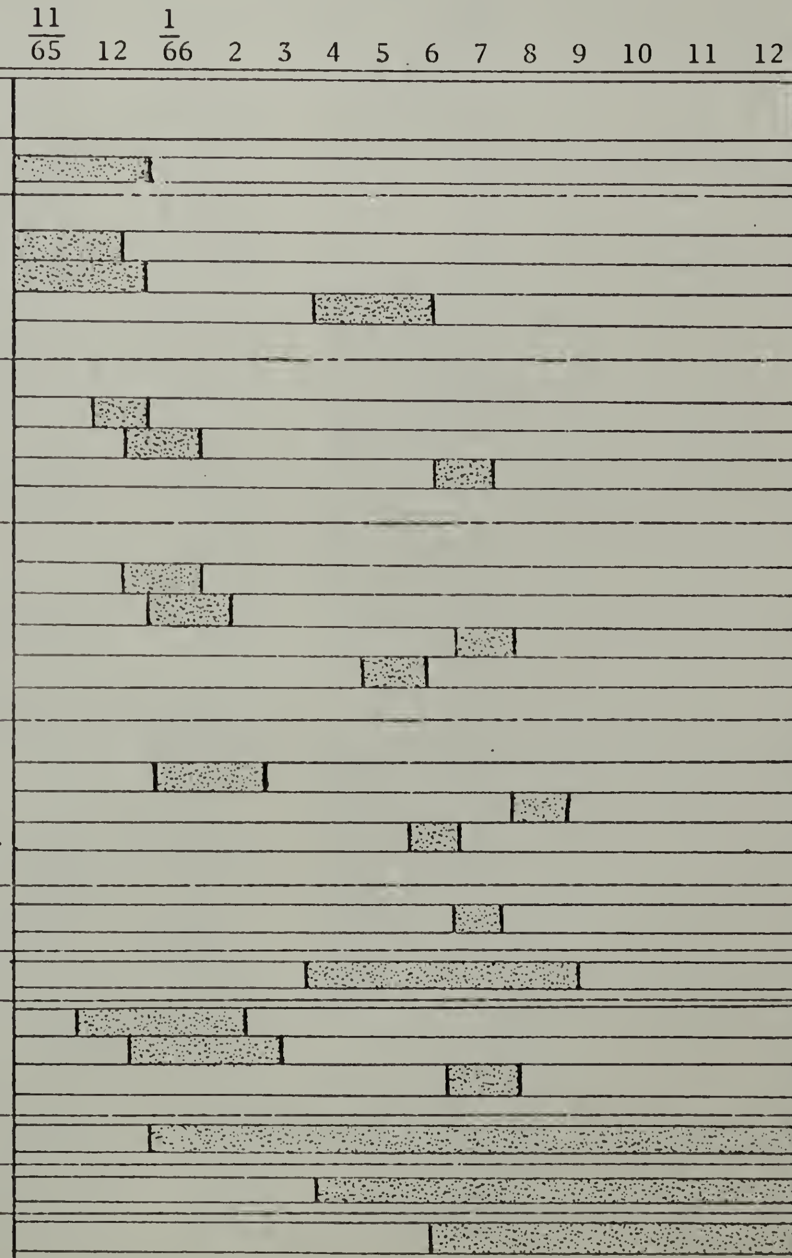
This report was prepared by the Corrosion Section, National Bureau of Standards under Contract No. H-2151A "Investigation of the Directional Effects in the Stress Corrosion of Aluminum Alloys" for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the technical direction of the Propulsion and Vehicle Engineering Laboratory, Materials Division of the George C. Marshall Space Flight Center with D.B. Franklin acting as project manager.

## *PROGRAM PLANNING CHART*

| PROGRAM STAGES | TECHNIQUES | MATERIAL |
|---|---|---|
| Literature Search | | |
| Material Procurement | | Extrusion<br>Plate<br>Replacement Plate |
| Macroscopic Studies | Metallographic Surveys | Extrusion<br>Plate<br>Replacement Plate |
| Tensile and Disk Specimen Preparation | Miniature Spec.<br><br><br>Subminiature Spec. | Extrusion<br>Plate<br>Replacement Plate |
| Tensile Properties | Notched and Unnotched Tensile Tests | Original Material<br>Replacement Plate<br>Subminiature Spec. |
| Construction of Constant Temperature S.C.* Apparatus | | |
| Susceptibility Evaluation | S.C. Test & Metallography | |
| Effect of Morphology | Optical and Electron Metallography | Extrusion<br>Plate<br>Replacement Plate |
| Effect of Plate Depth | S.C. Tests and X-Ray Studies | |
| Effect of Rolling Direction | S.C. Tests and X-Ray Studies | |
| Role of Segregation | Microprobe Analysis | |




*S.C. - Stress Corrosion

## ANTICIPATED WORK

A. Continue preferred orientation studies which will relate slip plane orientation to susceptibility.

B. Determine reason for deviation of 7075-T651 plate surface specimen from predicted susceptibility.

C. Extend the study of the role of segregation and its relationship to stress-corrosion cracking.

## ABSTRACT

This period's work has been concerned with three areas. The first, a study of stress-corrosion susceptibility as a function of specimen orientation, was carried out to check the testing environment for its simulation of accelerated atmospheric conditions. The second and third were direct attacks on the role of directionality in the stress-corrosion susceptibility. The second area was a study of preferred orientation as a function of both depth and direction. The third was electron probe studies showing the relation of segregation of various alloy constituents to direction.

## WORK ACCOMPLISHED TO START OF CURRENT REPORTING PERIOD

The work accomplished up to the start of this current period was concerned with providing the necessary data to carry out the directionality studies. It consisted of the procurement of materials, study of the microstructure of the material, production of appropriate specimens, mechanical testing, stress-corrosion testing and determination of preferred orientation. In detail, this work can be listed as follows:

1) *Material Procurement:* Delay was encountered due to the manufacturers recall of the 2219 plate material which was found to be defective. Prior to the recall, considerable work was done on this material.

2) *Structure Studies:* Macroscopic examination was made of all materials to determine areas from which specimens were to be removed. The microstructures of the various alloys were also examined so that grain shape might be related to stress-corrosion susceptibility. A preliminary electron microscope study was also made of an etched surface of the 7075-T6 alloy extrusion.

*3) Production of Specimens:* Both notched and standard round tensile specimens were machined from the extruded material and standard round tensile specimens were also machined from the plate material. Specimens were machined from each material in three orientations with respect to the rolling direction. Disc shape specimens were also machined from various levels parallel to the rolling plane from all plate material for preferred orientation studies.

*4) Mechanical Testing:* Tensile tests were completed for all materials to verify the specified mechanical properties. The effect of notch radius on mechanical properties of the extruded material was also studied.

*5) Stress-Corrosion Testing:* Environment--Total immersion in Alcoa H solution (0.3% NaCl, 3.0% $K_2Cr_2O_7$, 3.0% $CrO_3$, solution pH 0.9) was found to be superior to anodic polarization in 3 1/2% NaCl. The relationship between stress-corrosion susceptibility and specimen orientation was established for all materials (except the 2219-T37 plate material) and found to be as expected. Subsequent tests are to be made on the 2219-T37 plate material.

*6) Preferred Orientation Studies:* A study of the relationship between preferred orientation and distance from the surface of plate material was begun.

## WORK PROGRESS

The work accomplished previous to this period has been concerned with gathering data that furnishes a foundation upon which the studies for this period are based. These studies have been concerned with three areas. The first, a study of stress-corrosion susceptibility as a function of specimen orientation was carried out to check the testing environment for its simulation of accelerated atmospheric conditions. The second and third were direct attacks on the role of directionality in the stress-corrosion susceptibility. The second area was a study of preferred orientation as a function of both depth and direction. The third was electron probe studies showing the relation of segregation of various alloy constituents to direction. Both the second and third studies were not completed during this period, but significant results, as described in the following sections, were obtained.

## STRESS-CORROSION SUSCEPTIBILITY VERSUS SPECIMEN ORIENTATION

Constant temperature apparatus has been assembled so that all stress-corrosion tests can be carried out at a constant temperature of 35.0 ± 0.1°C. This will eliminate the effects of temperature variation on

susceptibility. Figure 1 shows the flow of the recirculating corrosive solution from the constant temperature reservoir and coil to the cell containing the stress-corrosion specimen. The system holds approximately 250 ml of solution which is discarded after each specimen is tested. Failure times are recorded to the tenth minute with an interval timer activated by a microswitch placed beneath the weights used to stress the specimen. When the specimen fails, the solution is automatically drained to prevent excessive corrosion of the specimen after failure.

Short transverse specimens of 2219-T87 and 7075-T73 alloy plate were tested at a constant stress of 75% of their yield strengths in Alcoa H solution (.30% NaCl, 3.00% $K_2Cr_2O_7$, 3.00% $CrO_3$, solution pH 0.9). The results are given in Table 1.

Metallographic investigation of failed specimens of the 2219-T87 and 7075-T73 showed that both types of material underwent both pitting and stress-corrosion cracking. The 2219-T87 exhibited primarily intergranular corrosion with little pitting while the 7075-T73 showed intergranular cracks extending from pits. However, with both types of material there was a great deal of variation in the amount of pitting and of intergranular corrosion from one specimen to another, which might account for the range in the endurance times.

Stress-corrosion studies are to be completed for all plate material in each of the three orientations.

## PREFERRED ORIENTATION VERSUS SUSCEPTIBILITY OF 7075-T651*

Preferred orientation studies were completed on 7075-T651 Aluminum alloy plate. Flat disc specimens were machined from various levels in a 2.5 inch thick plate. Pole figures of the (111) reflex were determined for specimens at 0.26, 0.57, 0.73, 0.89, and 1.20 inches below the surface of the plate. These pole figures are shown in Figure 2a through 2e, respectively. The relative intensities shown are an indication of the number of slip planes oriented with their poles at the angles shown on the pole figures.

*Role of Depth for a Given Direction:* For short transverse stress-corrosion specimens, the stress direction is as indicated in Figure 3. The highly susceptible grain boundaries, i.e., those normal to the stress direction, are in the equator plane. If the (111) pole is at an angle of $\phi$ with the equator plane (grain boundary), then the (111) plane is at an angle of $90 - \phi = \theta$ with the grain boundary. It is then seen in Figure 2 that the $\theta$ values for highest intensity of (111) reflections change from 25-45° near the surface to 55-70° at the center of the plate.

Robertson and Tetelman[1] have indicated that susceptibility of f.c.c.

*We wish to acknowledge the many helpful discussions concerning this phase of the project with Dr. L.P. Skolnick of the University of Maryland

alloys undergoing intergranular corrosion is greatest when the (111) are oriented at 70° to grain boundaries which are normal to the stress direction as shown in Figure 4. The basis for this was theory by Stroh[2] which says that the normal stress $\sigma$ acting on the grain boundary* making an angle $\theta$ with the slip plane is:

$$\sigma = 3/2\,(L_o/r)^{1/2}\ \sigma_o \sin\theta \cos 1/2\,\theta$$

where $\sigma_o$ = applied stress

$L_o$ = length of slip plane occupied by a group of piled-up dislocations

$r$ = crack length

$\theta$ = angle between slip plane and grain boundary.

Assuming $L_o$, $r$ and $\sigma_o$ constant, it can be seen that the normal stress acting on the grain boundary (or probability of cracking) is proportional to $\sin\theta \cos 1/2\,\theta$. ($\sigma_o$ was varied slightly in our tests, but not enough to account for the differences in susceptibility.)

Figure 5 is a plot of $\sin\theta \cos 1/2\,\theta$ against $\theta$ (the angle between slip planes and the grain boundaries. It is seen that the probability of cracking is greatest at 70°. Also shown on this graph are plots of average intensities of (111) vs. $\theta$ for the specimens taken from various levels within the plate. Average intensities for five degree angle ranges were taken from the X-ray data because of the highly fluctuating nature of that data. Figure 5 clearly indicates that stress-corrosion susceptibility should increase with distance from the surface of the plate.

To see if this relation between depth and susceptibility exists as indicated by the preferred orientation studies, stress-corrosion tests were run on subminiature tensile specimens, dimensions shown in Figure 6, machined from various depths of a 2.5 inch thick plate of 7075-T651 Aluminum alloy. These were all electropolished to reduce the effects of machining. All but 1/8" of the gage length was covered with a protective rubber coating. Six (five in one case) specimens from each location were tested under constant load at 50% of yield strength in the Alcoa H solution. All specimens were tested at a constant temperature of 35°C, using the constant temperature equipment of Figure 1.

Failure times for each level in the plate are given in Table 2. The arithmetic mean of the failure times is given as is also a mean calculated

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*Only grain boundaries normal to the applied stress are assumed important.

from the logarithms of the failure time. The mean based on the logarithm of endurance is believed to be a better way of comparing the endurance time because the logarithm of endurance is normally distributed as shown by Booth and Tucker[3].

Figure 7 shows the log mean endurance time and the range in data plotted against distance from plate surface. A statistical analysis of variance was performed on the data. At the 0.01 significance level, the conclusion was that the data were not from a homogeneous population. Therefore, the groups of specimens did differ significantly in the variability of their endurance times (i.e., the times to failure for the different groups were significantly different.)

On comparing the stress-corrosion results with the mechanical properties shown in Figure 1 of the Quarterly Status Report for the quarter ending July 31, 1966, it can be seen that the mechanical properties cannot account for the differences in the stress-corrosion endurance at the various levels on the plate. It was seen there that the mechanical properties remained nearly constant except at 3/8" from the surface, where the tensile properties were considerably higher.

However, on comparing the stress-corrosion results with the preferred orientation results, Figure 8, it is seen that the preferred orientation predicts susceptibilities as observed (i.e., more susceptible toward the center) for all but the specimens taken from 3/8" below the surface of the plate. Figure 12 shows observed susceptibility as (endurance time)$^{-1}$ and predicted relative susceptibility plotted against distance from the surface of the plate. It is expected that the curve for observed susceptibility would be of the same shape as that for predicted susceptibility if more specimens were tested between the 1 1/8 and 1 3/8" levels.

It is also believed that the group of specimens from 3/8" depth were more susceptible due to the stress level at which they were run. Since all specimens were run at 50% of Y.S., and the 3/8" level specimens had the highest yield strength, they were run at the highest level.

*Role of Direction for a Fixed Depth:* Besides the effect of depth for a given direction, further examination of Figure 2 shows that at a given depth (the plate interior) the preferred orientation results would predict the susceptibilities as: short transverse > long transverse > longitudinal. It can be seen that for the short transverse stress direction (normal to plane of pole figure) the intensity of reflections from favorably oriented (111) is greatest and for the longitudinal stress direction (same as rolling direction shown) the number of faborably oriented (111) planes is least. For the long transverse stress direction (in the plane of the pole figure, but normal to the rolling direction), the number of faborably oriented (111) plane is seen to be between that for the short transverse and longitudinal directions. Therefore, the

number of favorably oriented slip planes might be used to predict the relationship between susceptibility and orientation. Whether this prediction holds or not will be tested in future work.

## THE ROLE OF SEGREGATION IN THE SUSCEPTIBILITY OF 7075

Electron probe microanalyses were made of 7075-T651 and -T73 material. Specimens were taken perpendicular to the rolling plane from the center and from near the surface of the plate. At each level, specimens were taken both perpendicular and parallel to the rolling direction.

The target current images of areas with inclusions are shown in Figure 9 for the specimens near the surface of both the 7075-T6 and -T73 conditions. The light areas indicate inclusions of lower atomic number and the dark indicates inclusion of higher atomic number than the matrix. In Figure 10, the magnesium X-ray images, the light areas are high in magnesium content. These areas correspond to the light particles in Figure 9 and as determined by other X-ray images, are also high in silicon. It was also determined that the dark inclusions of Figure 9 were low in magnesium and silicon.

Figure 11, the iron X-ray images, shows iron to be highly segregated at the dark particles of Figure 9. Copper and manganese were also segregated at these inclusions. Aluminum and zinc were found to be depleted at all inclusions.

The chromium X-ray image, Figure 12, shows that there are zones depleted of chromium that do not correspond to the inclusions of Figure 9.

Titanium, not shown, also follows the chromium. These depleted zones are seen to be more prominent in the 7075-T651 than in the -T73 material and are believed to correspond to subgrain boundaries and may explain the higher susceptibility of 7075-T651. Further investigation will be made to try and correlate these depleted zones directly with the grain or subgrain boundaries.

The only difference found in the electron probe microanalysis between the material from the center and from near the surface of the plate was that approximately twice the magnification was required for the specimens from near the surface in order to see inclusions of the same size as near the center. This is believed to be due to the more highly deformed nature of the material near the surface. The significance of this segregation of different constituents as a function of directionality will be explored more deeply by the electron probe technique in future work.

## REFERENCES

1. Robertson, W.D. and A.S. Tetelman, Strengthening Mechanisms in Solids, ASM, p. 217 (1960).
2. Stroh, A.N., Proc. Roy. Soc., 223, 404 (1954).
3. Booth, F.F. and G.E.G. Tucker, Corrosion, Vol. 21, p. 173 (1965).

TABLE 1

Endurance times of short transverse specimens in Alcoa H solution.

2219-T87

| Specimen No. | | Time (min.) |
|---|---|---|
| 104 | | 195.8 |
| 103 | | 215.6 |
| 102 | | 253.1 |
| 106 | | 325.1 |
| 108 | | 430.3 |
| 107 | | 454.2 |
| | Average | 312.3 |

7075-T73

| Specimen No. | | Time (min.) |
|---|---|---|
| 105 | | 401.6 |
| 104 | | 417.6 |
| 106 | | 563.4 |
| 103 | | 616.2 |
| 107 | | 650.6 |
| 102 | | 1072.3 |
| | Average | 620.3 |

TABLE 2

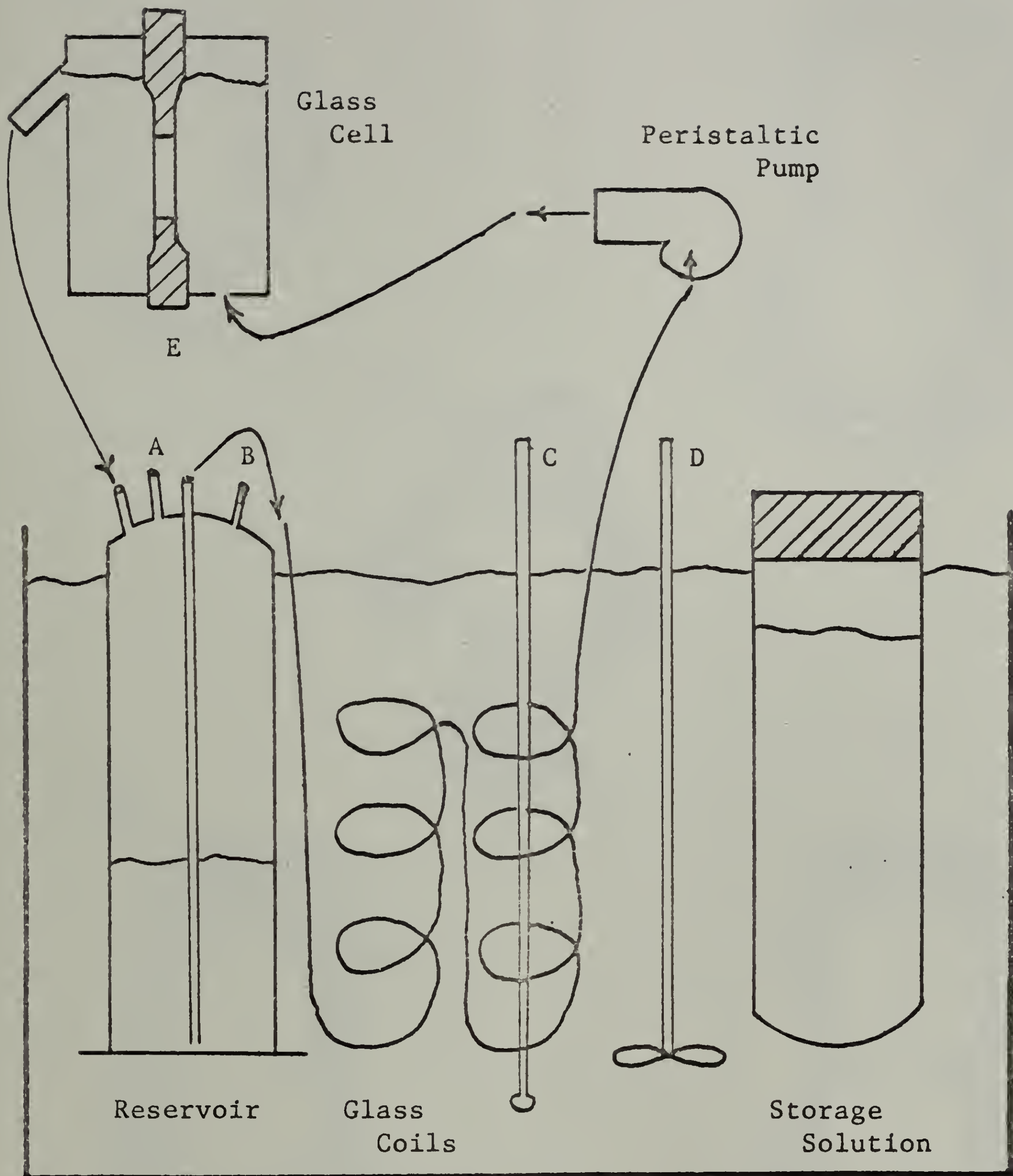
STRESS CORROSION ENDURANCE TIME AT VARIOUS LEVELS

IN A PLATE OF 7075-T651 ALUMINUM ALLOY

| Distance from Surface *(in.) | Mean Endurance Time (min.) | Log Mean Endurance Time **(min.) |
|---|---|---|
| 3/8 | 4.66 | 4.57 |
| 5/8 | 11.91 | 11.40 |
| 7/8 | 9.63 | 8.74 |
| 1 1/8 | 7.07 | 6.82 |
| 1 3/8 | 5.02 | 4.97 |

*Distance from surface is distance to center of 1/4" gage length specimens. Six specimens were run from all but the 3/8" level where five were run.

**Log mean endurance time is the anti-logarithm of the mean of the logarithms of the times to failure.




KEY

| | |
|---|---|
| A | Fill Vent |
| B | Fill Hole |
| C | Bath Thermometer |
| D | Stirer |
| E | Miniature Specimen |

Figure 1. Constant temperature stress-corrosion testing system showing flow of corrosive solution.

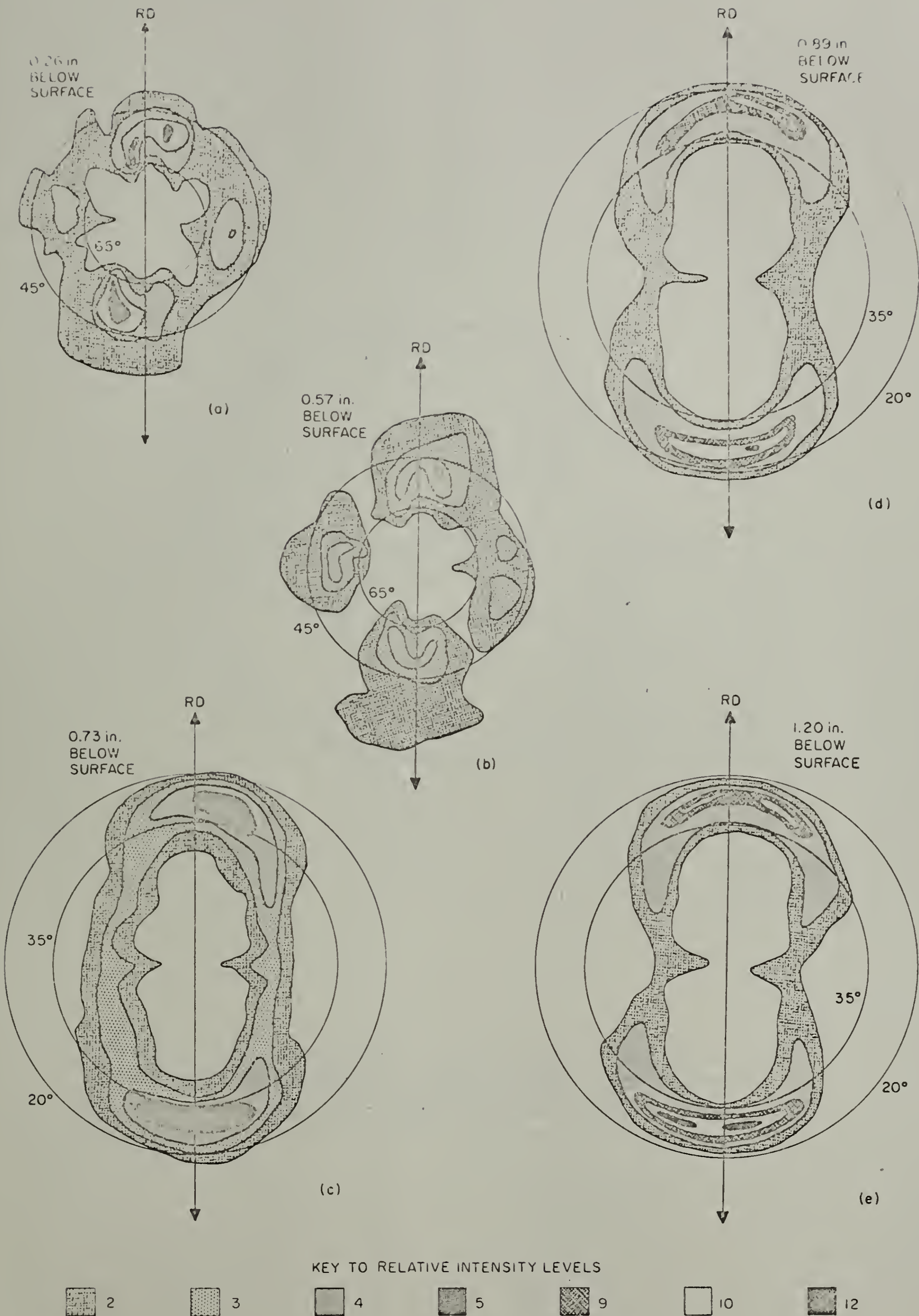



FIGURE 2: (111) - POLE FIGURES OF SPECIMENS PARALLEL TO THE SURFACE OF A 2.5 in. THICK 7075-T651 ALUMINUM ALLOY PLATE.

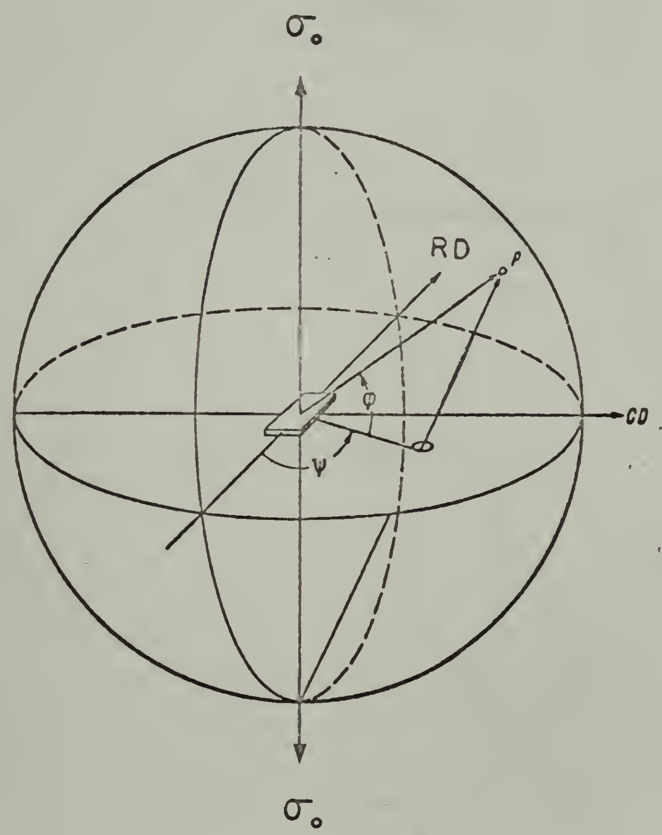



FIGURE 3: Representation of lattice planes of a rolled metal specimen in stereographic projection. The equator plane was chosen as the plane of projection.

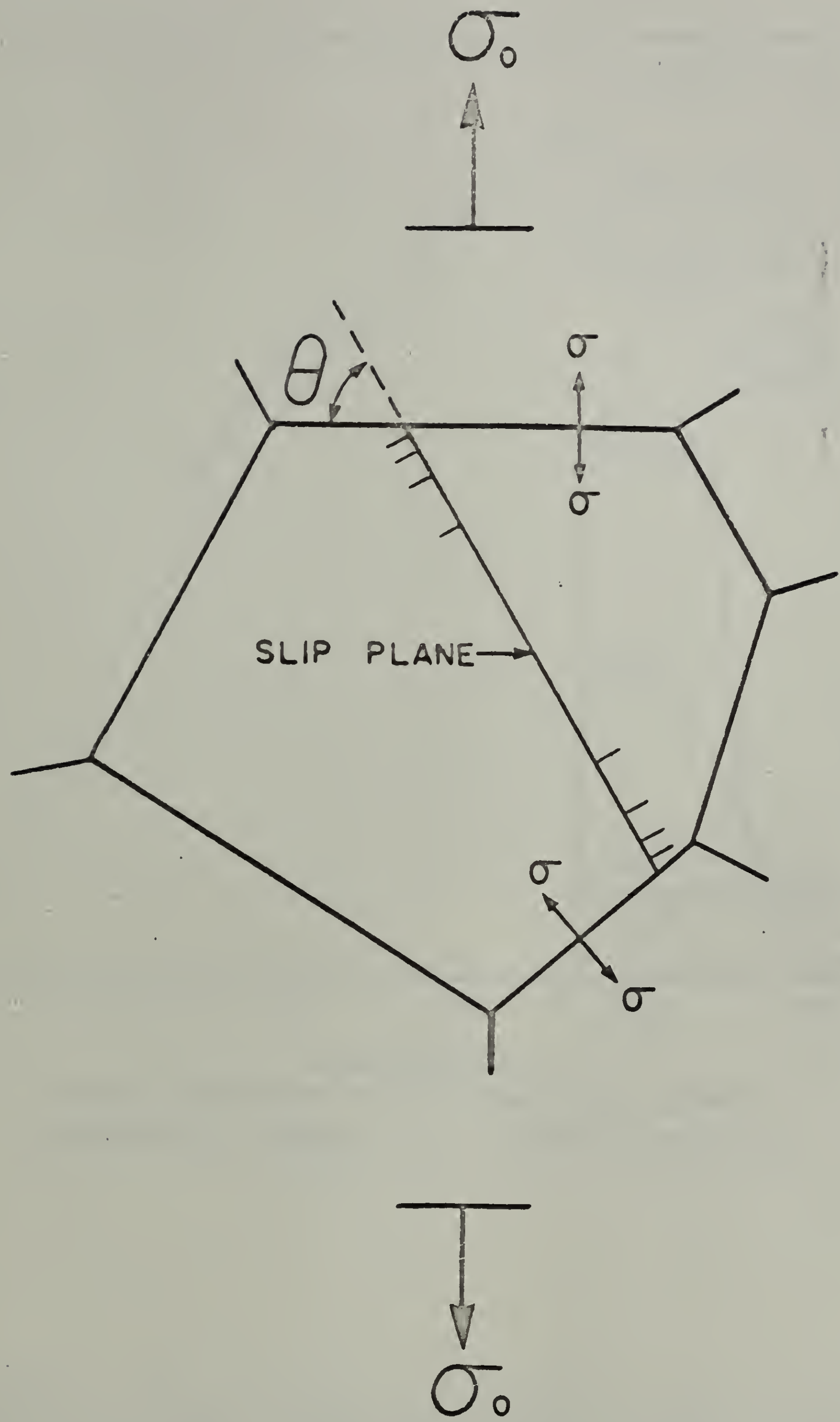



FIGURE 4: The stress normal to the grain boundary, $\sigma$, is maximum when both the grain boundary is normal to the applied stress and the stress due to dislocations piled up is greatest. The stress due to piled up dislocations is greatest when the slip plane is at an angle of $\Theta = 70.5°$ to the grain boundary.

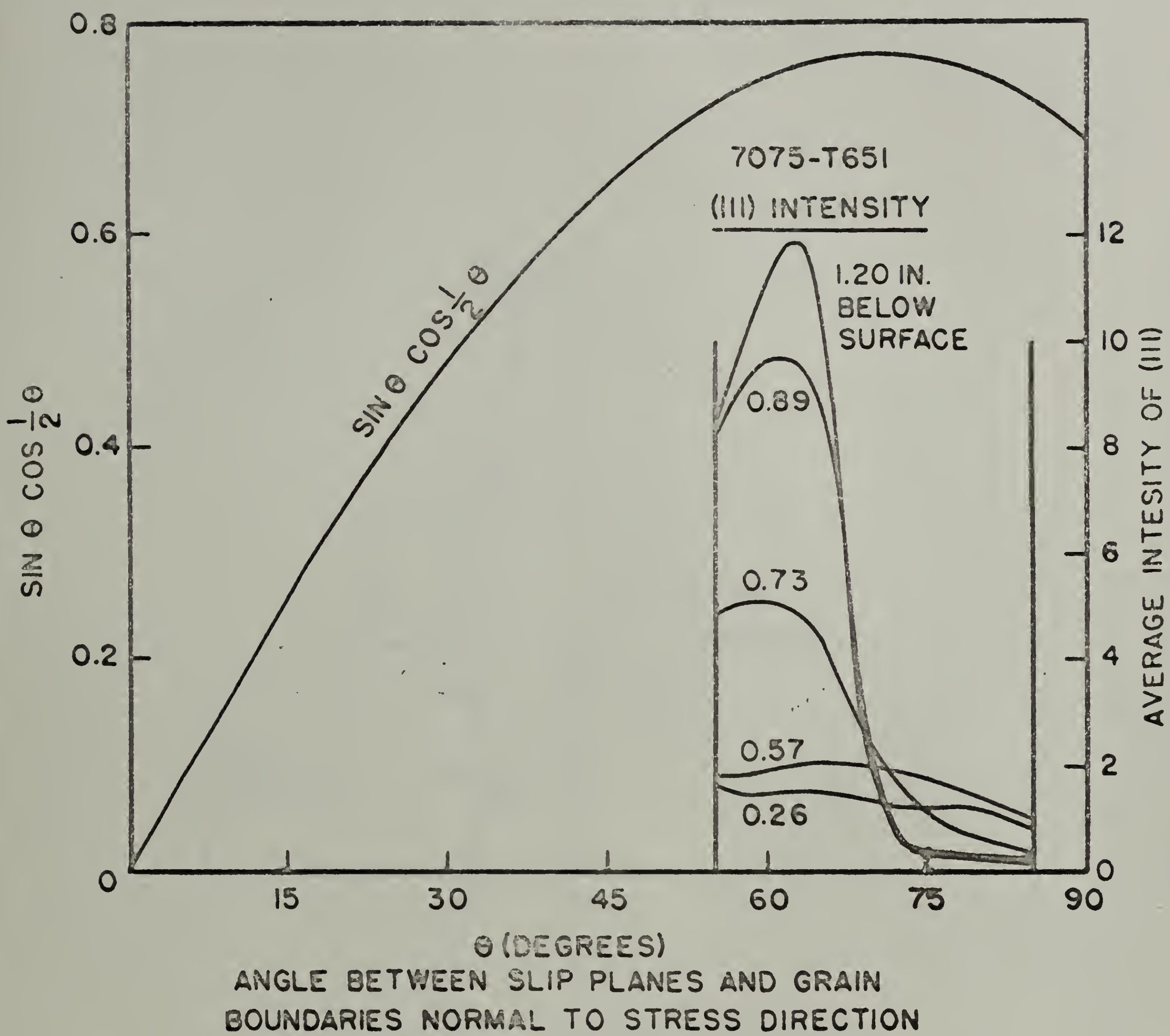



FIGURE 5: The probability of cracking is shown plotted against the angle between slip planes and grain boundaries normal to the applied stress. Superimposed on this are plots showing the intensity of (111) reflections at various depths in a 7075-T651 Aluminum alloy plate. The intensity of (111) reflections in the range from θ = 55 to 85° is a measure of the number of (111) favorably oriented for stress corrosion cracking.

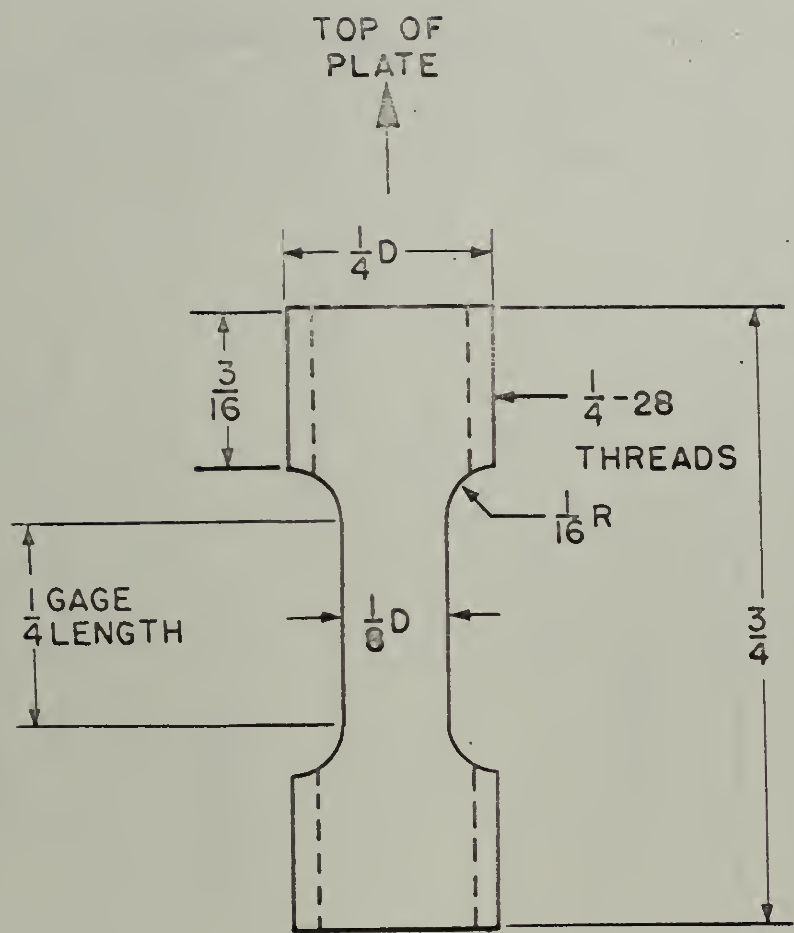



FIGURE 6: Dimensions of subminiature round tensile specimens used to determine the susceptible to stress corrosion cracking at various depths in a plate of material.

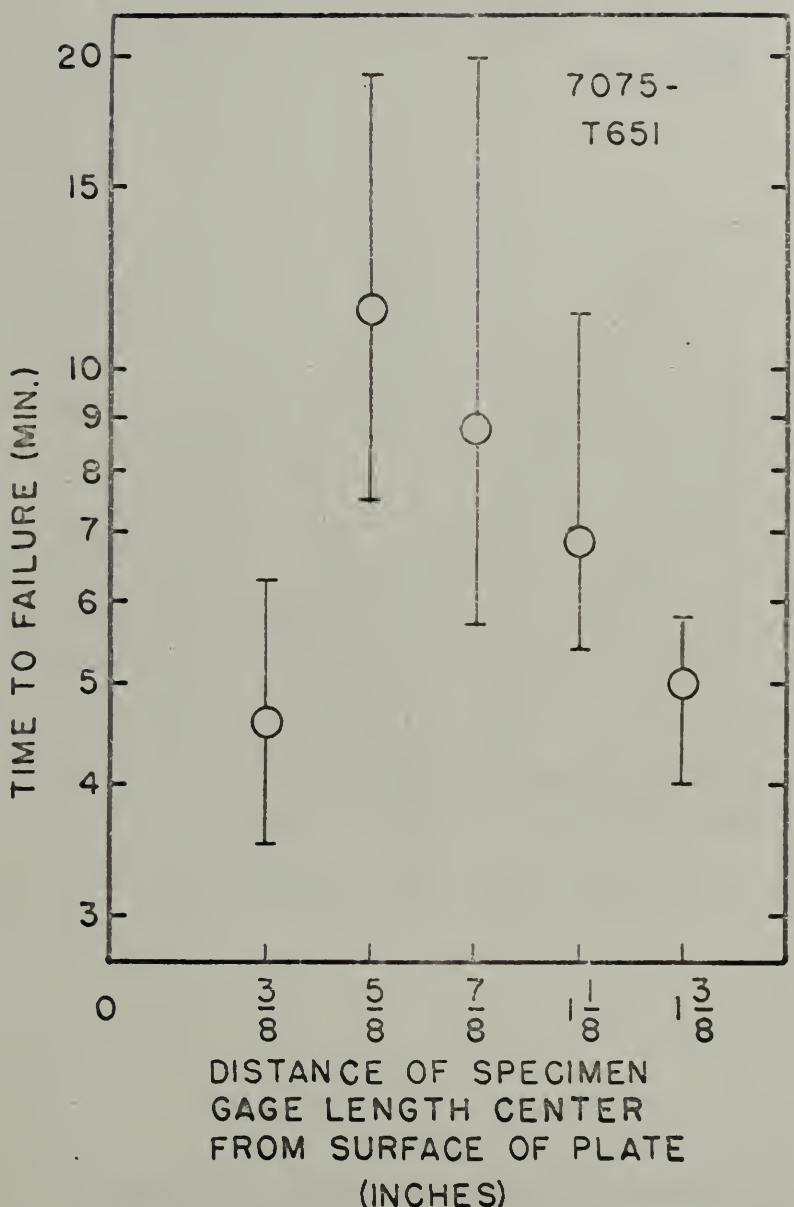



Figure 7: The stress-corrosion endurance of short transverse specimens is shown at various levels of a 7075-T651 Aluminum alloy plate. Both the log mean endurance time and the range in data are shown.

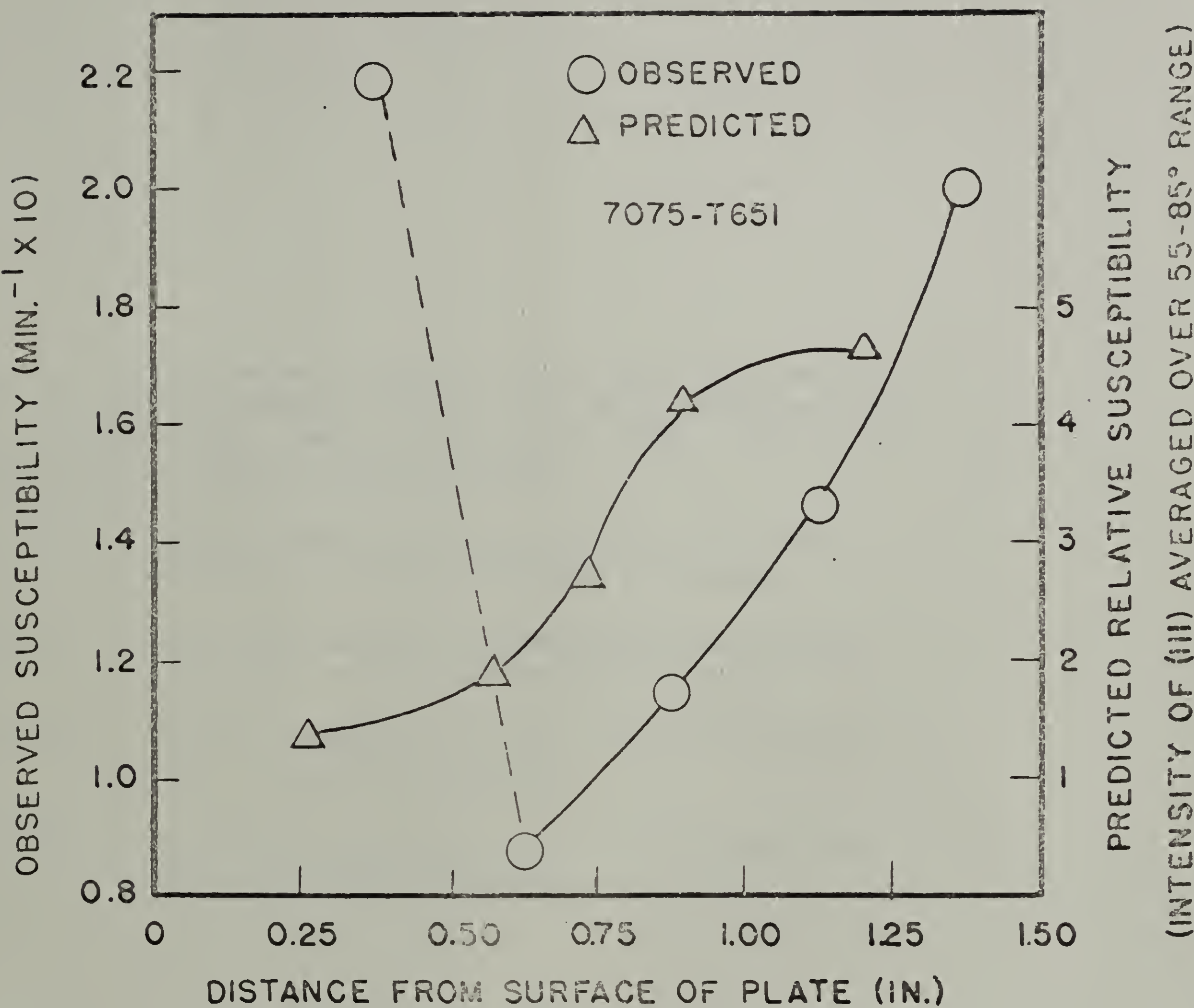
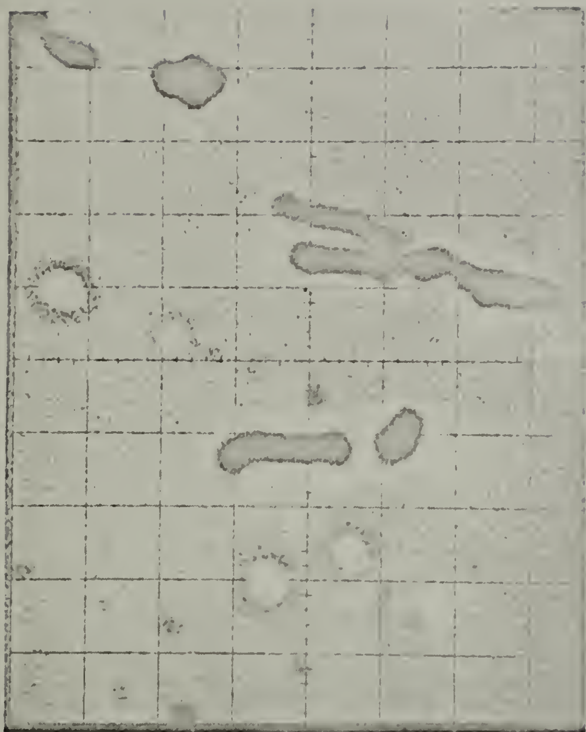


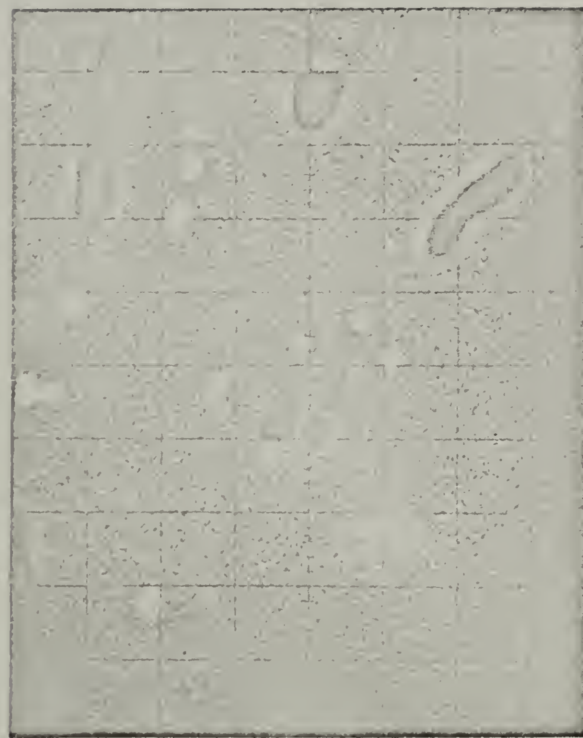

FIGURE 8: The observed stress corrosion susceptibility and the predicted susceptibility are shown plotted against distance from the surface of a 7075-T651 Aluminum alloy plate.

Figure 9. Target current images of areas of 7075 Aluminum alloy plate with inclusions. The light areas indicate inclusions of lower atomic number and the dark areas, inclusions of higher atomic number than the matrix.

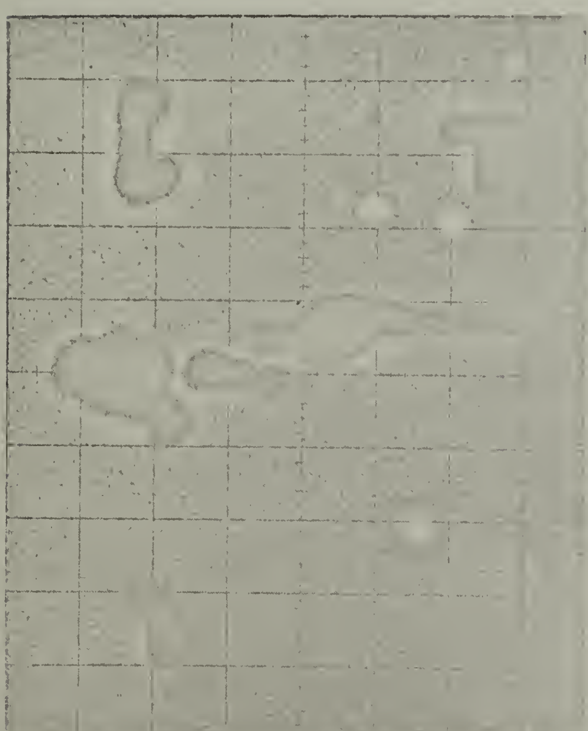
| | |
|---|---|
| T73-1S | 7075-T73 material perpendicular to rolling direction, at the surface of the plate. |
| T6-1S | 7075-T651 material perpendicular to rolling direction, at the surface of the plate. |
| T73-2S | 7075-T73 material parallel to rolling direction, at surface of plate. |
| T6-2S | 7075-T651 material parallel to rolling direction, at surface of plate. |



T6-1S



T6-2S



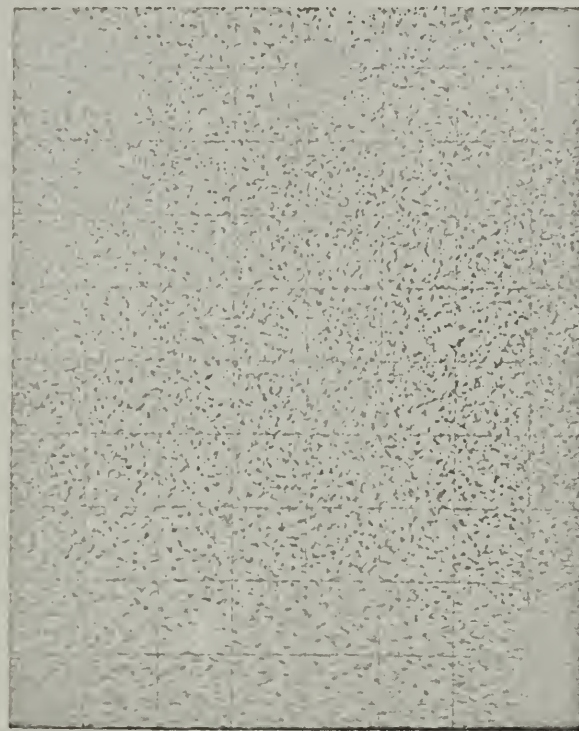
T73-1S

T73-2S

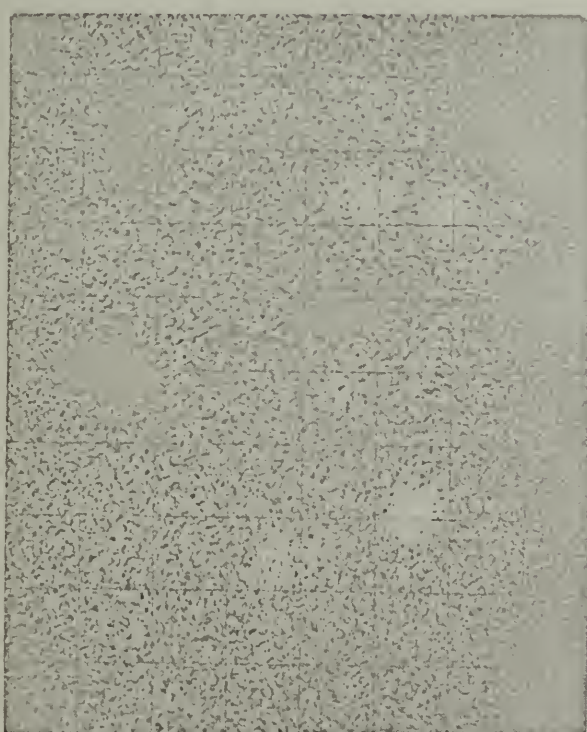
TARGET CURRENT IMAGES 7μ/div.

Figure 10. Magnesium X-ray images of areas shown in Figure 9. Light areas are high in magnesium.

T6-1S



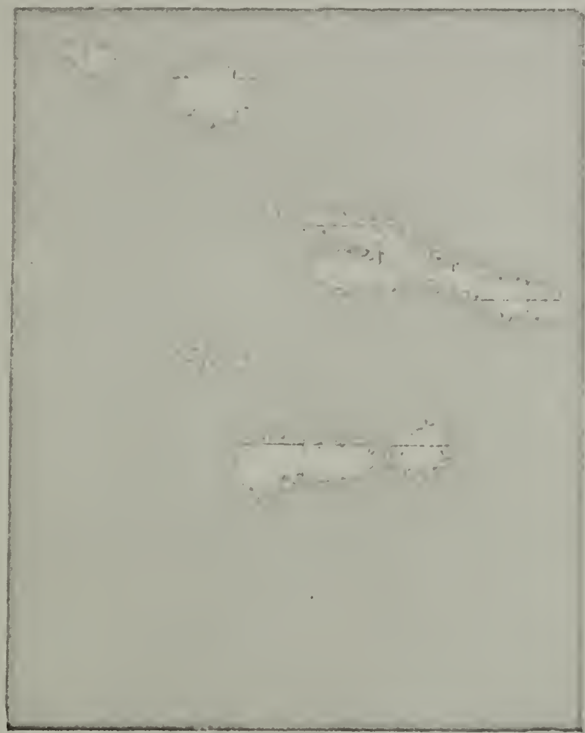
T6-2S



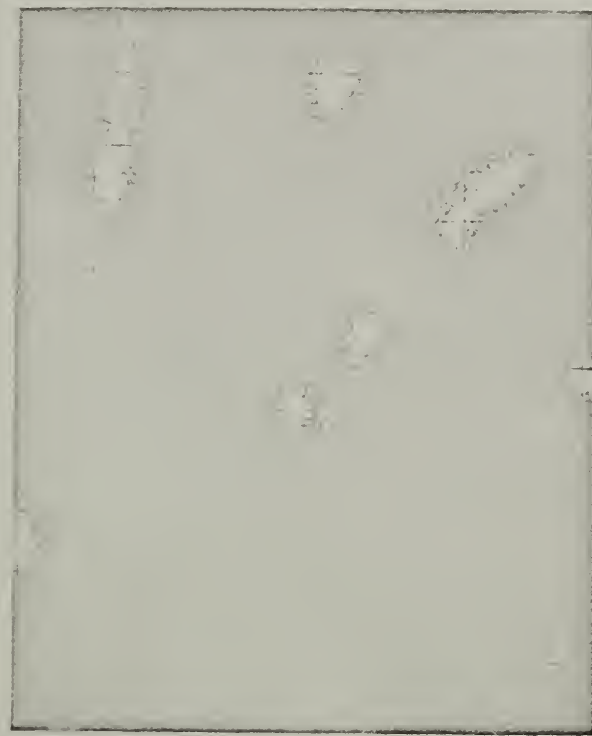
T73-1S

T73-2S

MAGNESIUM X-RAY IMAGES

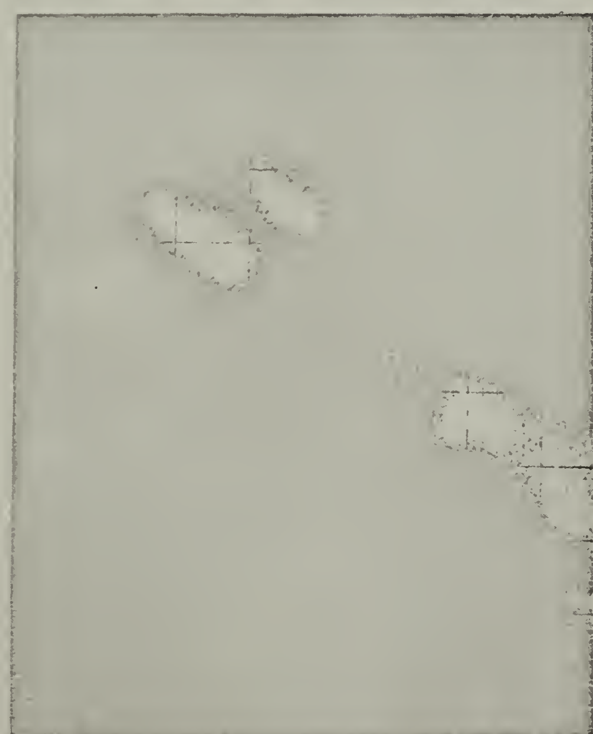


T6-1S



T6-2S

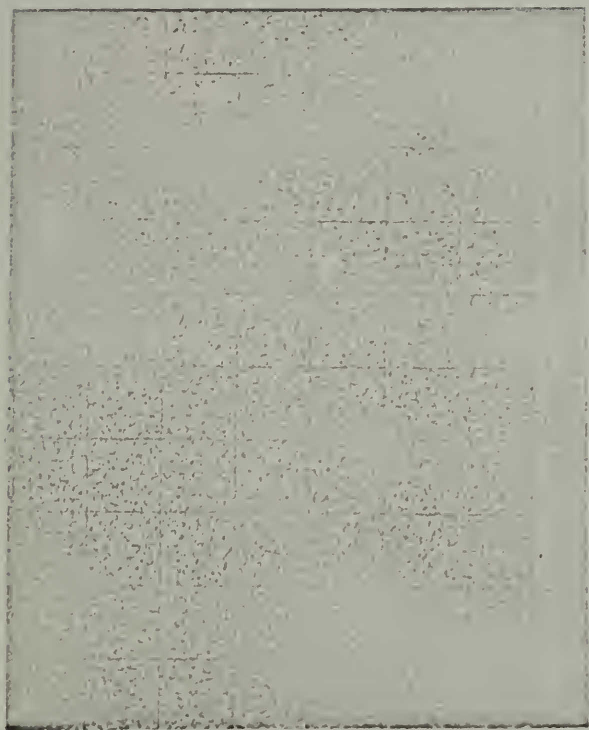
T73-1S



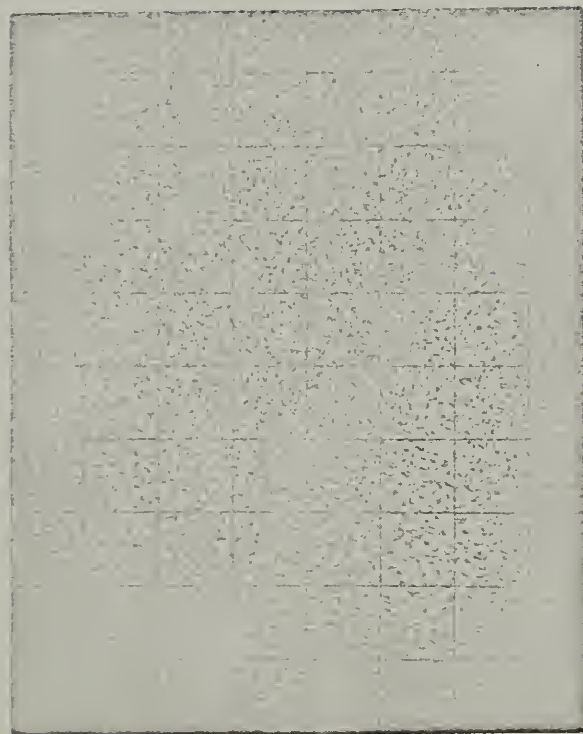
T73- 2S

IRON X-RAY IMAGES

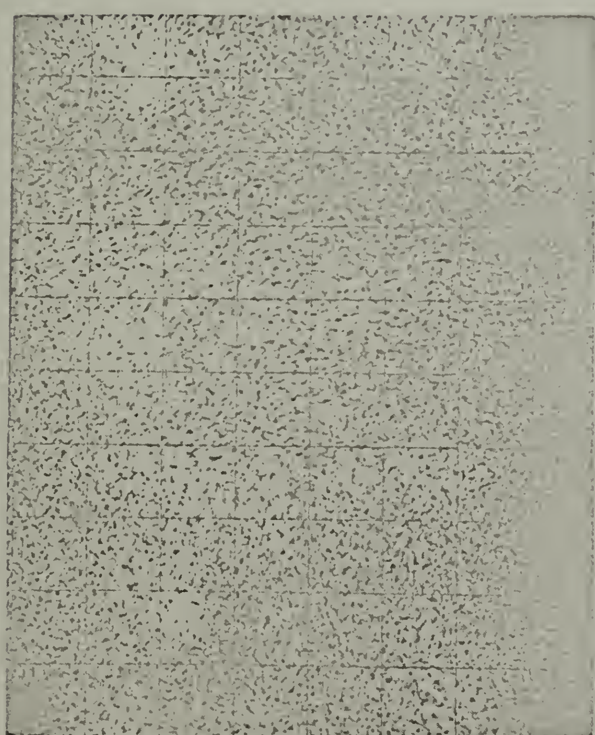
Figure 12. Chromium X-ray images of areas shown in Figure 9. Light areas are high in chromium.



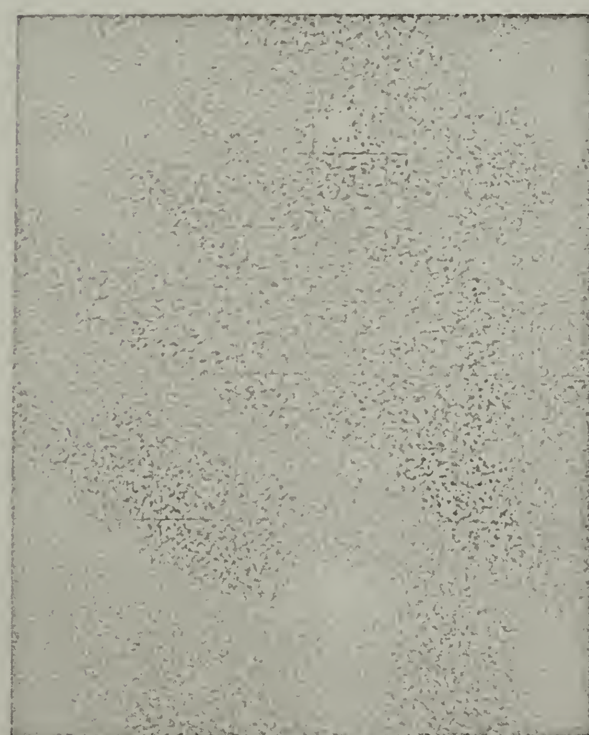
T6-1S



T6-2S



T73-1S



T73-2S

CHROMIUM X-RAY IMAGES